Tomographic Microscopy at the Swiss Light Source

M. Stampanoni

Swiss Light Source, Paul Scherrer Institut, CH-5232 Villigen, Switzerland

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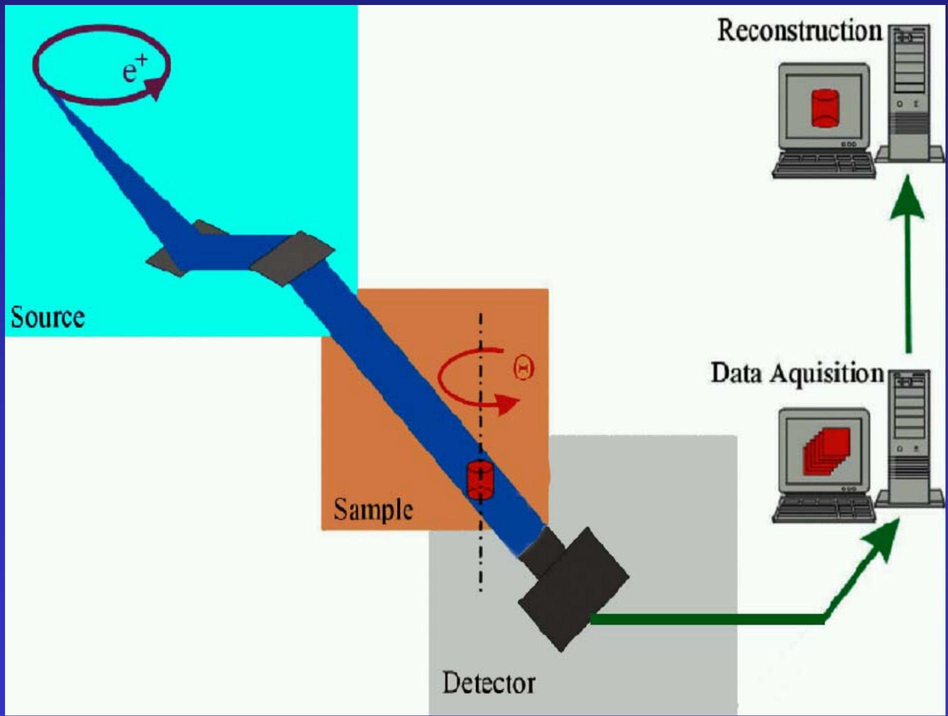
Synchrotron Based MicroCT: basic principle

X-ray computer tomography provides volumetric data of samples nondestructively by mapping their three dimensional x-ray attenuation.



Adapted from F. Beckmann, HASYLAB

Different detection methods:

- Optical magnification coupled to scintillating screens
  (SLS, ESRF, HASYLAB, NSLS, APS, ...)
- Zone Plates, Bragg Fresnel Lenses Compound Refractive Lenses
  (ESRF)
- Zooming Tubes
  (SPring8, only radiography)
- Bragg Magnifier
  (SLS)

Application fields

Research medium	Objects of interest	Required resolution
• Ceramics	Porosity, heterogeneity	≈ 1 µm
• Metal compounds	Defects, phase interfaces	≈ 1 µm
• Fibers	Defects, density fluctuations	≈ 1 µm
• Tissue engineering	Porosity, structure	≈ 1 µm
• Microelectronics	Cracks, grain bounderies	≈ 1 µm
• Medical bone research	Structure, mineralization	≈ 1 µm
• Biological tissues	Structure, composition	≈ 1 µm
• Geological rocks, ore	Structure, composition	≈ 1 µm

Need of intense X-ray flux

Photon density (ph/mm^2) for a given SNR:

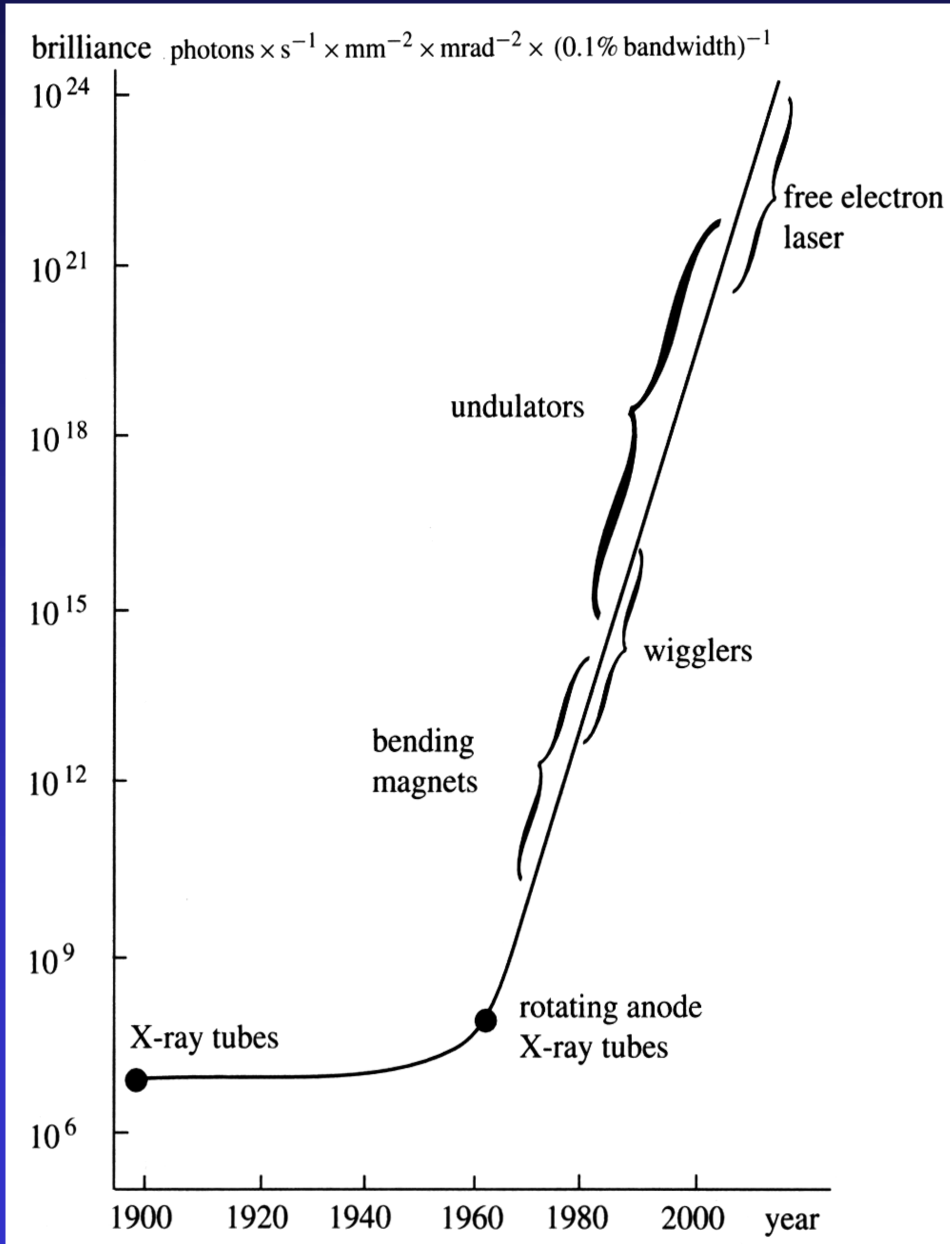
$$\Phi \propto \frac{SNR_{out}^2}{w^3 \cdot (\mu_1 - \mu_2)^2}$$

With:

- SNR = signal to noise ratio
- w = spatial resolution
- $(\mu_1 - \mu_2) =$ contrast signal

Further we know that:

$$w \Leftrightarrow DQE \equiv \frac{SNR_{out}^2}{SNR_{in}^2}$$



Winick, H., Synchrotron Radiation Sources, World Scientific Publishing, Singapore, 1995

Advantages of synchrotron radiation CT

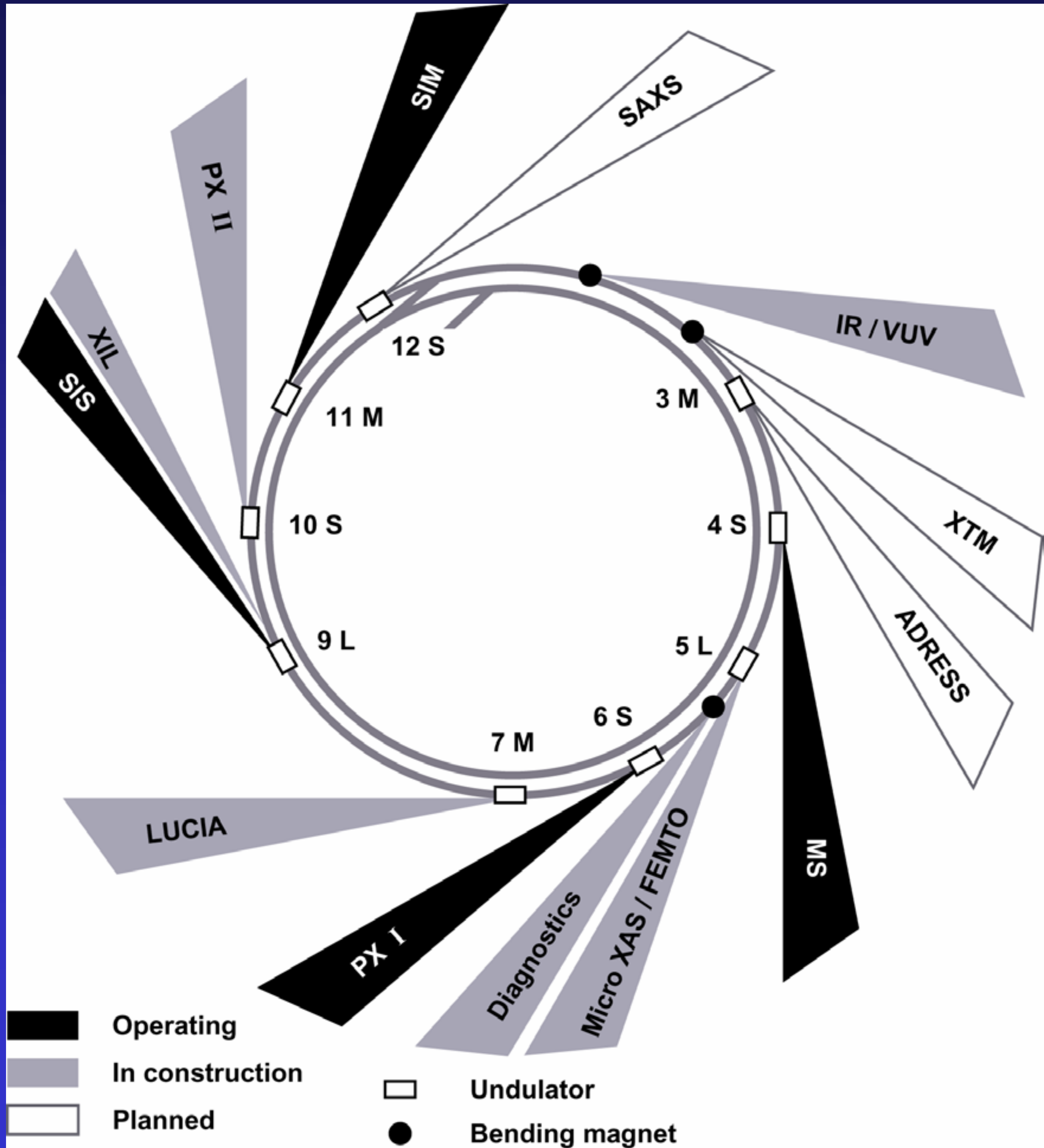
- The HIGH BRILLIANCE of the source enables high-resolution examinations in the micrometer range within reasonable exposure time

- The MONOCHROMATICITY eliminates beam hardening artefacts

- The ENERGY TUNABILITY makes element-specific measurements possible

- The COHERENT NATURE of the radiation extends absorption tomography to edge-enhanced and phase-sensitive investigations

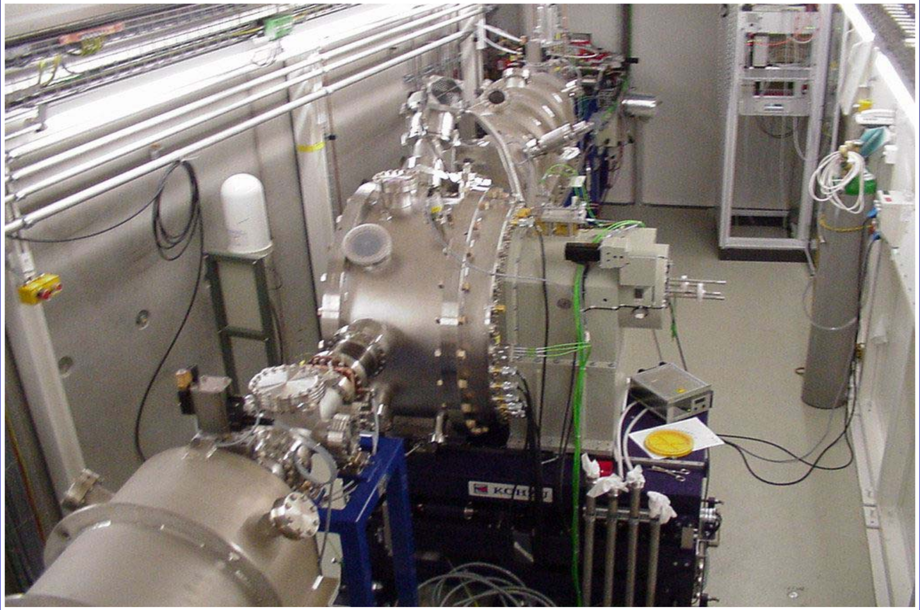
Swiss Light Source at PSI

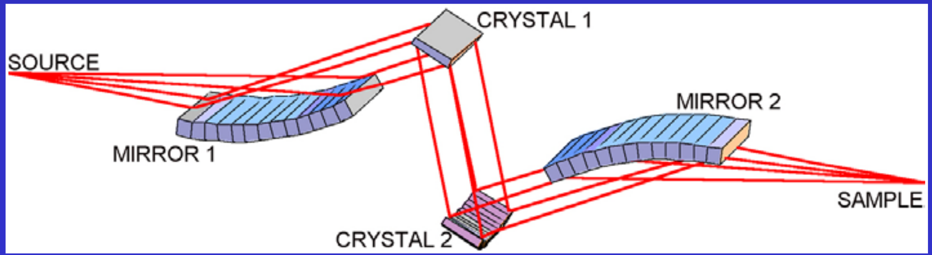


Materials Science Beamline 4S

Hybrid Minigap Wiggler (NdFeB)

- Energy range: 5 - 40 keV
- Flux : 10^{12} ph/(s mm^2 bw) at 20 keV
- Power : 8.4 kW
- 7.5 mm gap, 1.97 T, critical energy of 7.9 keV





X-ray optics

- First Rh-mirror for vertical collimation and harmonic suppression
- Fixed-exit double crystal Si(111) monochromator with energy resolution of 0.014%
- Second Rh-mirror for vertical focusing
- Horizontal focusing obtained by bending the second monochromator's crystal

Objectives of the SLS tomography device

The SLS detector should provide:

- Spatial resolution in the range of 5 micrometers to 500 nm
- High efficient photon detection with fast, low-noise readout
- Both absorption and phase contrast imaging
- Sample size up to several mm

SLS-XTM detector strategy

Standard Detector

Spatial resolution of 1 micron

State-of-the-art performances

YAG:Ce scintillator
1 to 50 microns

Microscope Optics
4x,8x,20x,40x

High Performance
Read-Out Electronics
2k x 2k, 14 bit, 250 ms

Submicrometer resolution

Novel instrumentation

Asymmetically cut Si(220) crystals

Structured CsI(Tl) scintillator

1:1 optic

Novel detector

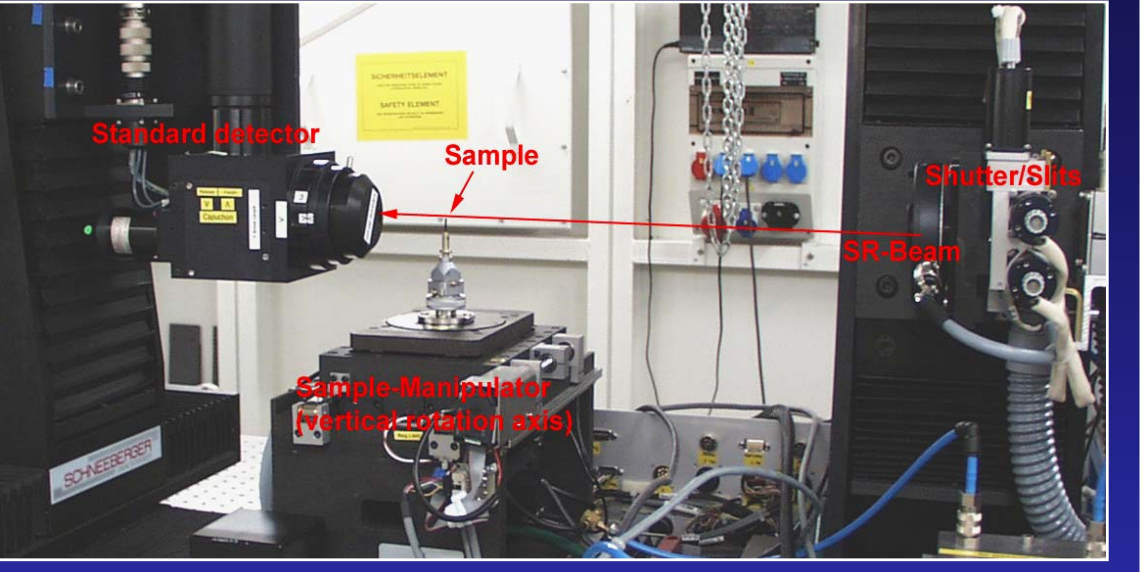
Tomography device at the 4S beamline

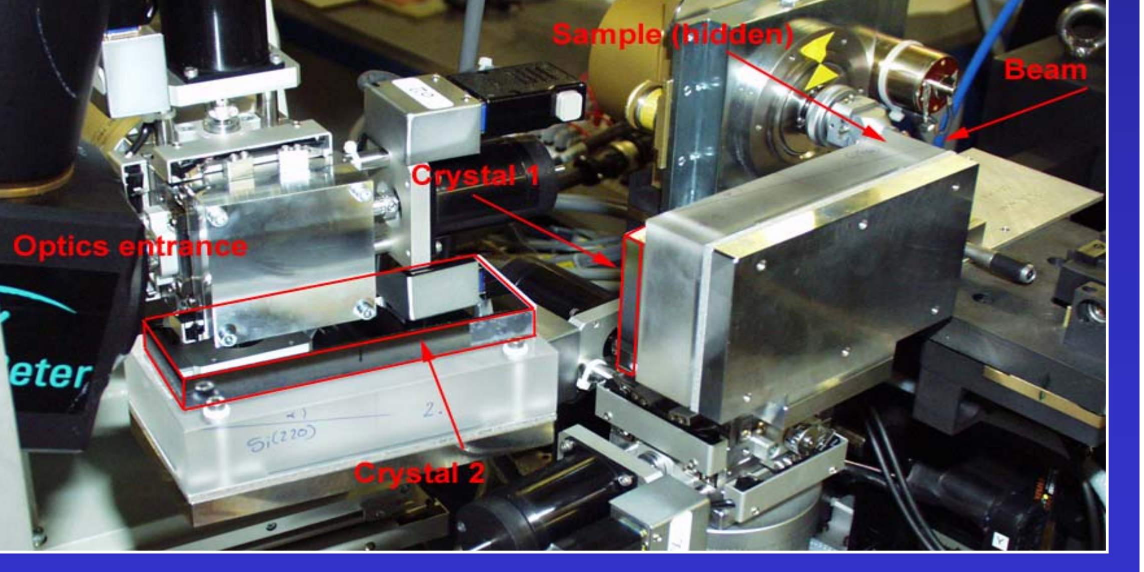
BEAMLINE KEYPOINTS
- Hybrid Minigap Wiggler (NdFeB)
- Si(111) double crystal monochromator
- Energy range: 5 - 40 keV
- Flux : 10^{12} ph/(s mm^2 bw) at 20 keV
- Horizontal and vertical focusing

STANDARD DETECTOR
- Magnification: 4x – 40x
- Spatial resolution : 1 – 5 µm
- Quantum efficiency: 7% at 20 keV
- CCD 2048x2048, 14 bit, 250 ms read-out

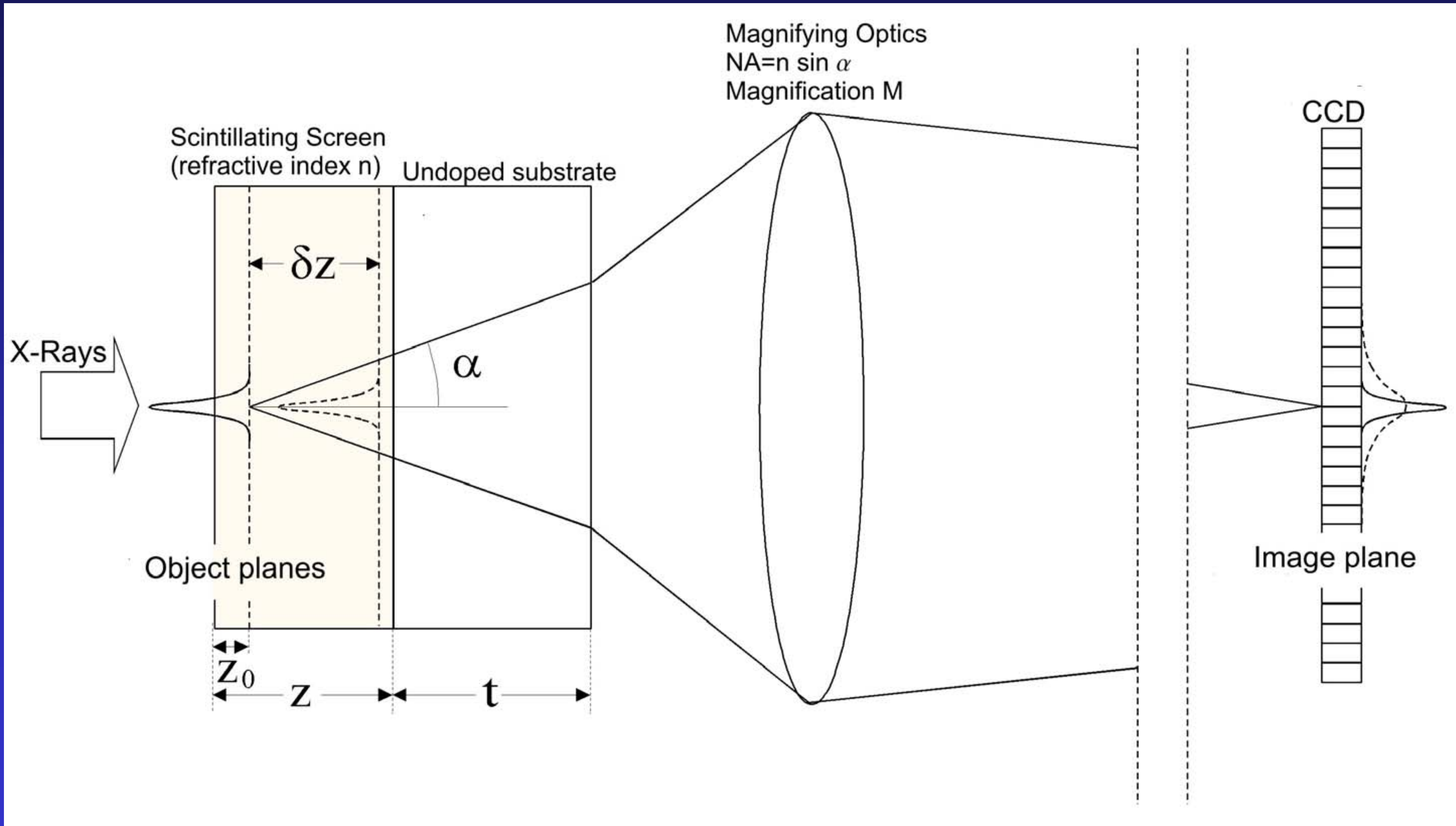
BRAGG MAGNIFIER
- Magnification: 10x – 100x
- Pixel size: 140-1400 nm
- Quantum efficiency: > 90% at 20 keV
- CCD 2048x2048, 14 bit, 250 ms read-out

SAMPLE HANDLER
- Wobble < 5 µrad, runout < 0.1 µm
- Pitch, roll: range ± 2°, resolution < 50 µrad
- In-out of beam with reproducibility <0.1 µm





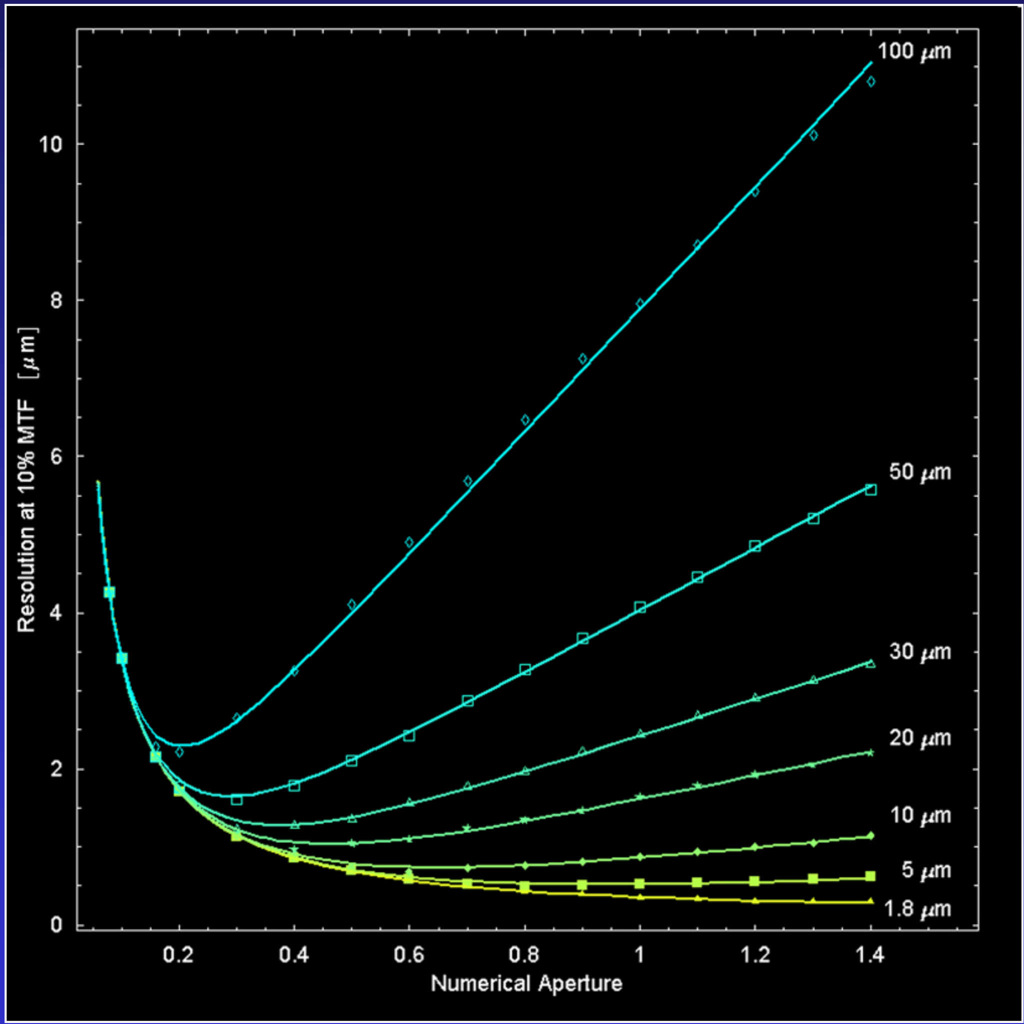
Working principle of the standard detector



SLS

Simulation of the detector frequency response

$$MTF(f) = \left|OTF(f, z, NA)\right| = \left|\int_{-z/2}^{z/2} D(f, \delta z, NA) \cdot d\delta z\right|$$



$D(f, \delta z, NA) =$ frequency response

$NA =$ numerical aperture

$\delta z =$ defect of focus

$f =$ frequency variable

Fitting function:

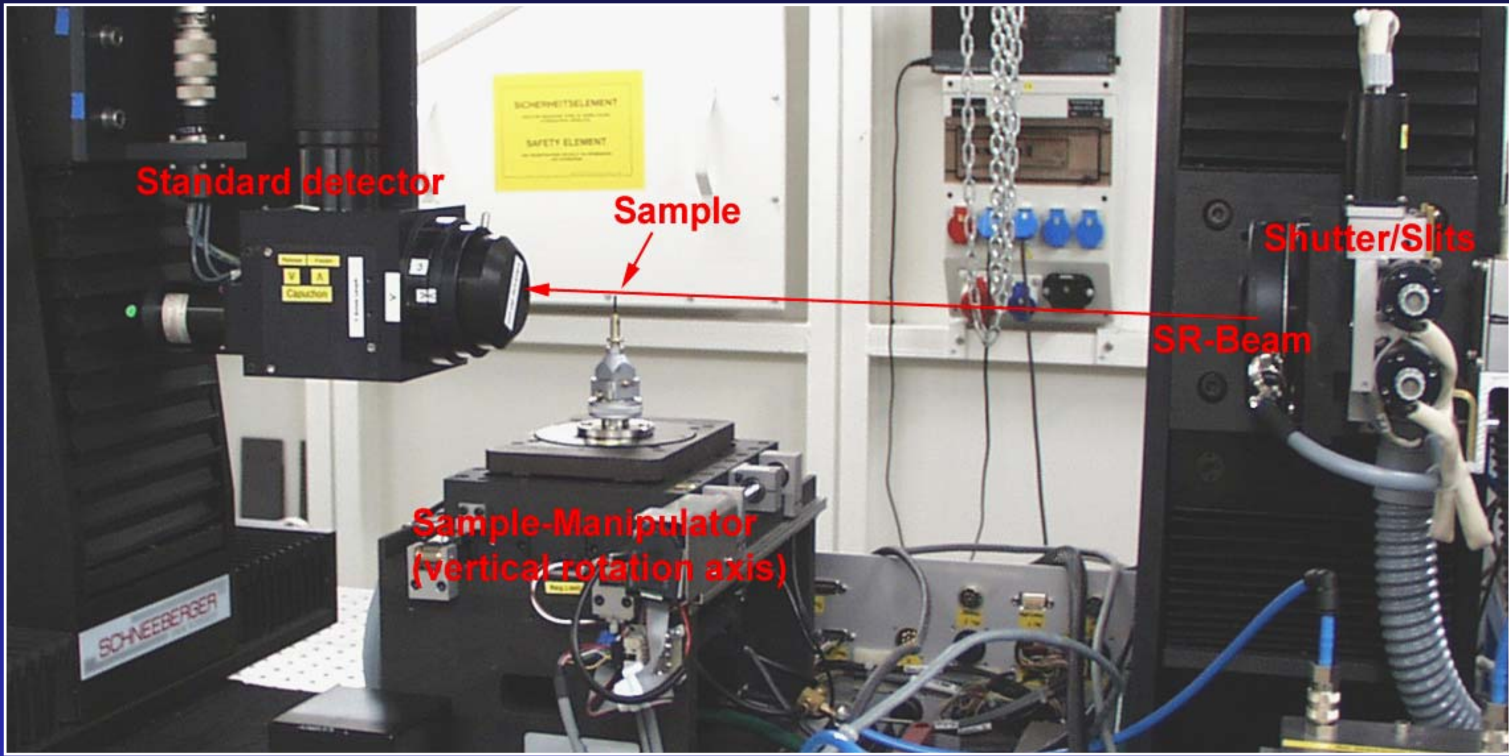
$$R = \sqrt{\left(\frac{p}{NA}\right)^2 + (q\ \ z\ \ NA)^2} \quad \text{with} \quad \begin{cases} p = 0.34\ \mu\text{m} \\ q = 0.036 \end{cases}$$

Diffraction

Defect of focus

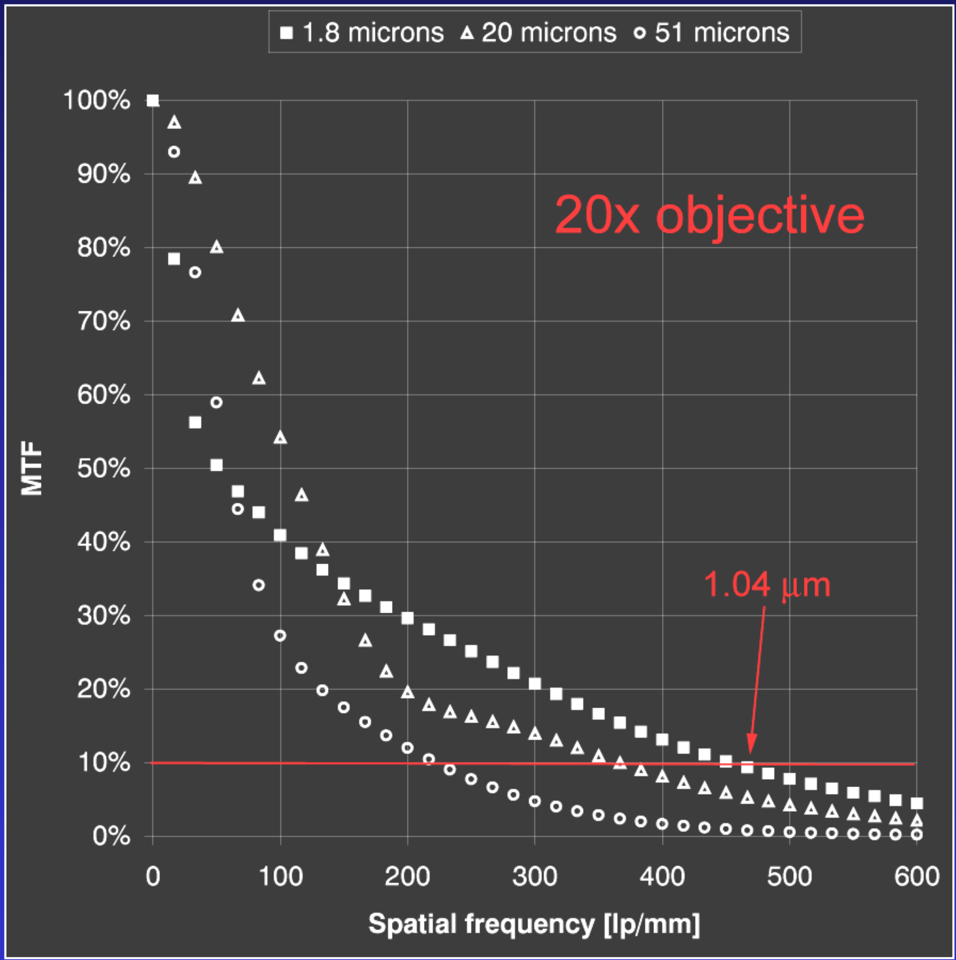
M. Stampanoni *et al.*, NIM A491 (2002), pp. 291-301

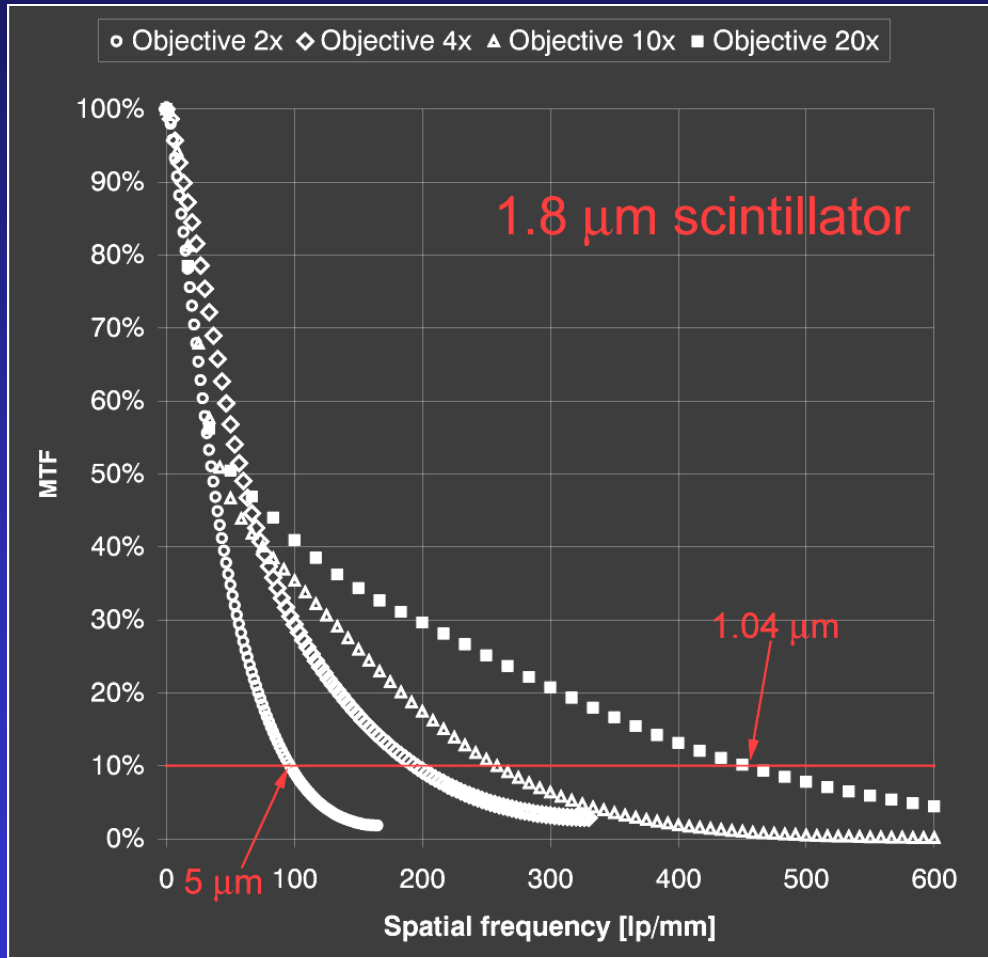
Changing the scintillator and the magnification...



SLS

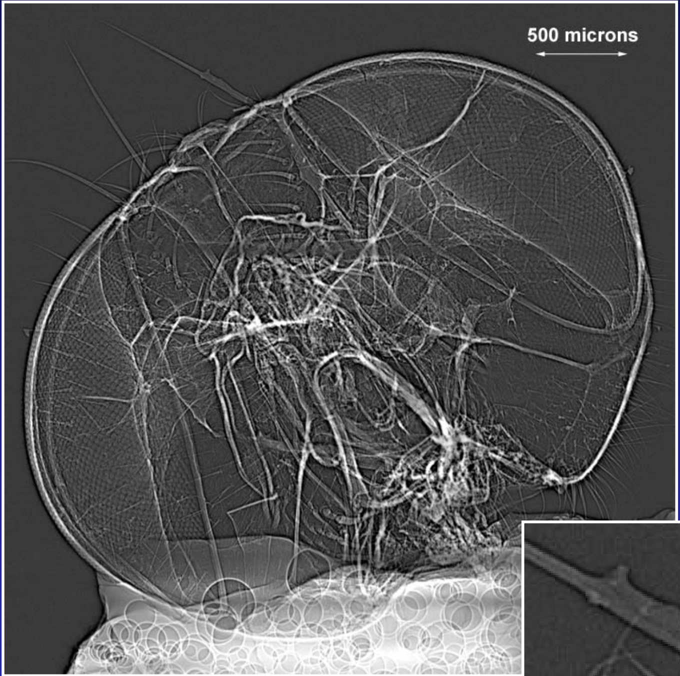
Measurement of the detector frequency response





M. Stampanoni *et al.*, NIM A491 (2002), pp. 291-301

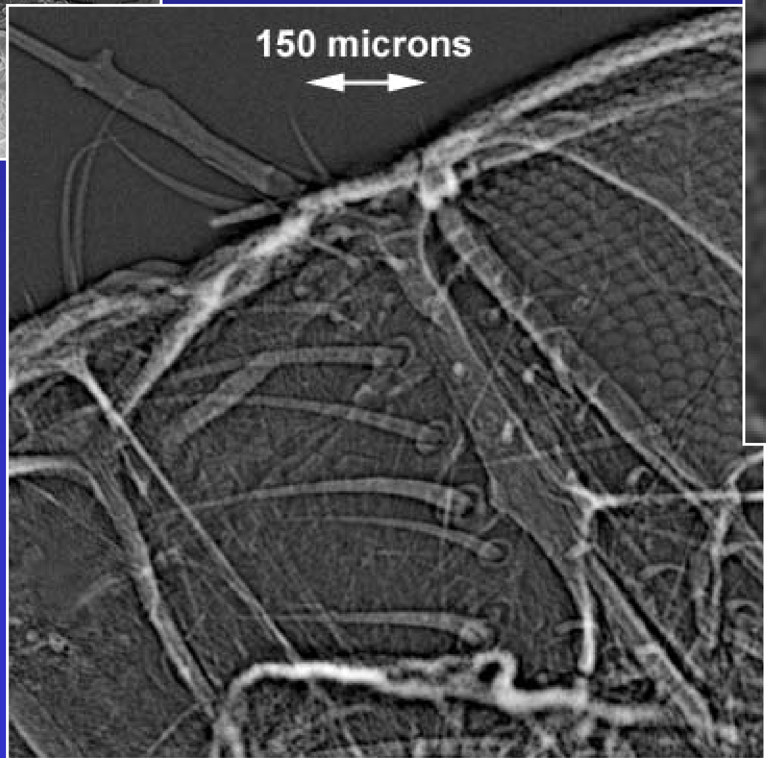
House Fly (first SLS picture, september 2001!)



Energy 9 keV

SDD = 100 mm

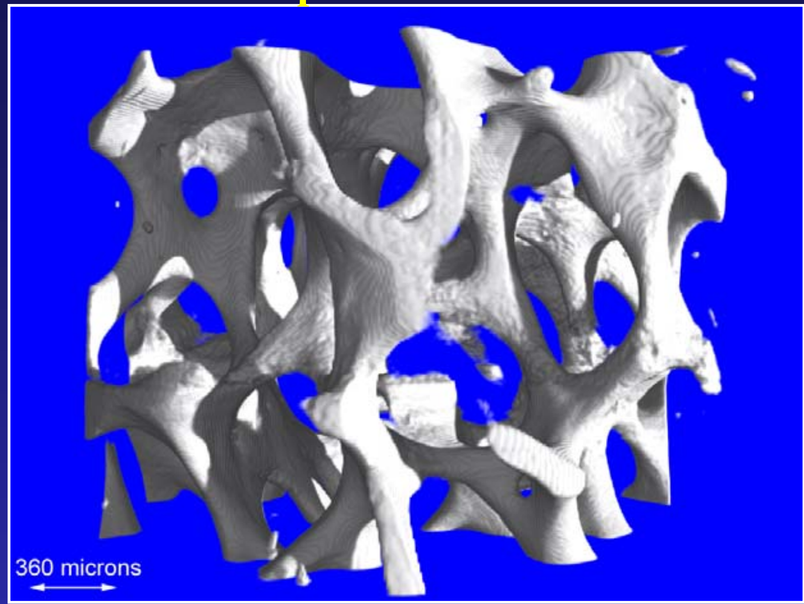
20 mm gap, 18.5 mA



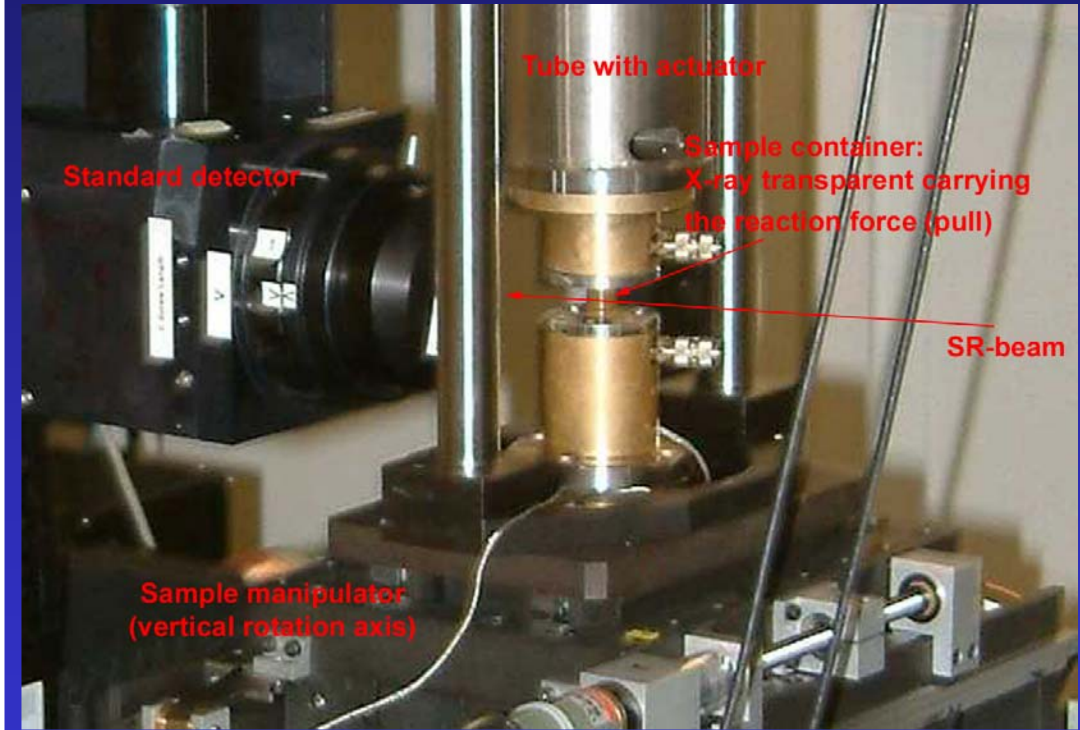
Assessment of bone sample damage

Courtesy of P. Thurner, EMPA and IBT

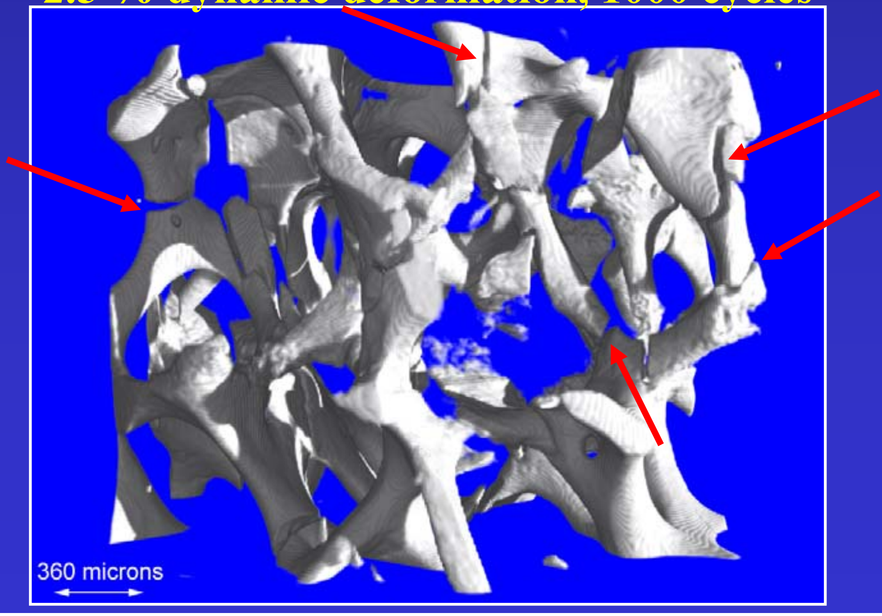
Sample before load.



Microcompression device installed on the sample holder.

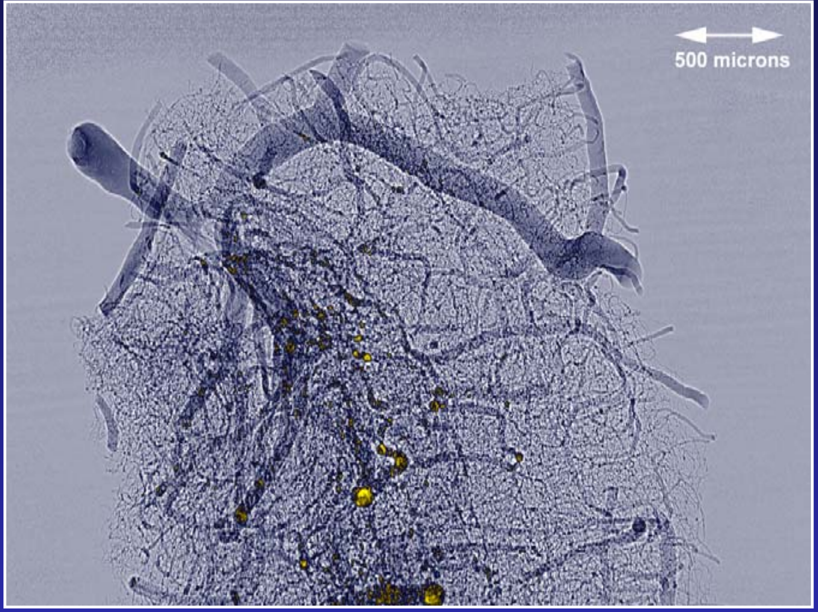


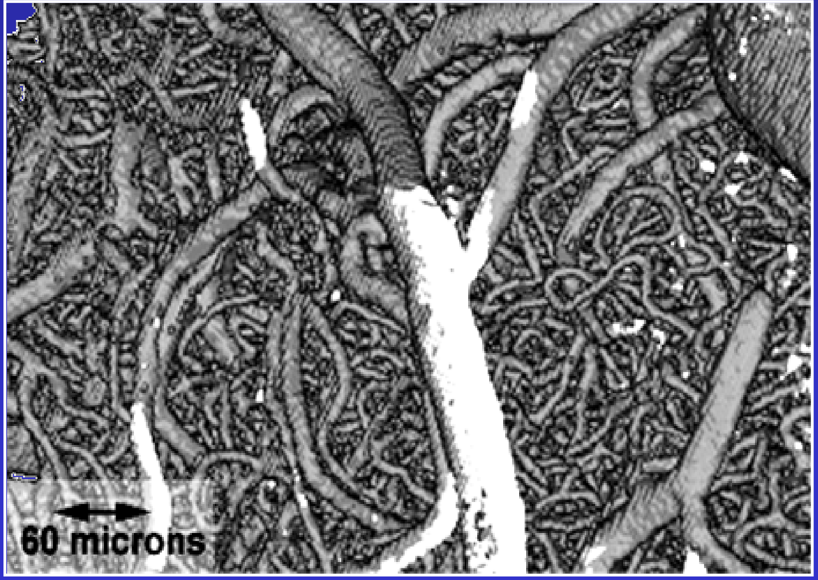
Sample after 5% static compression and 2.5 % dynamic deformation, 1000 cycles

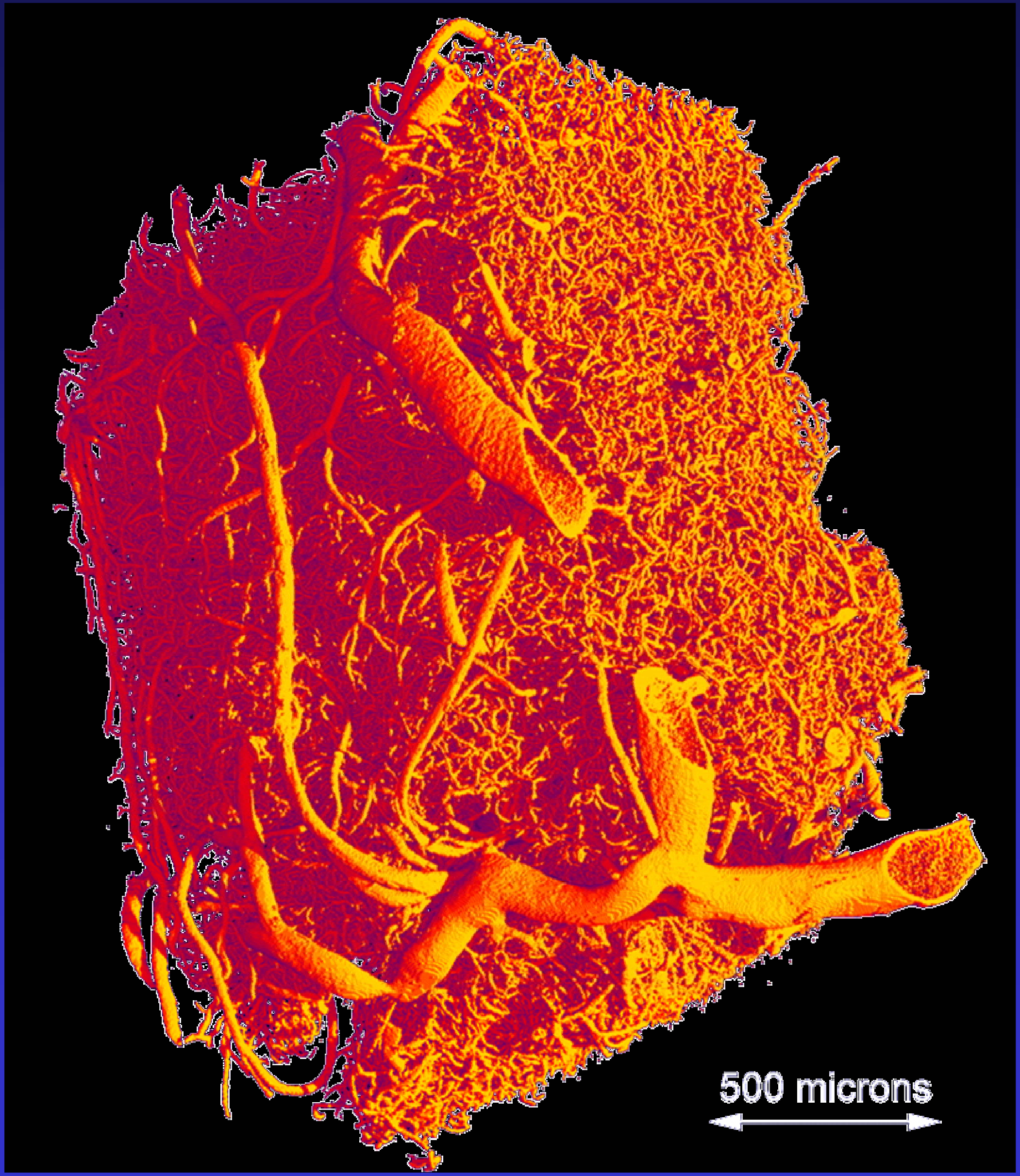


Alzheimer's disease research

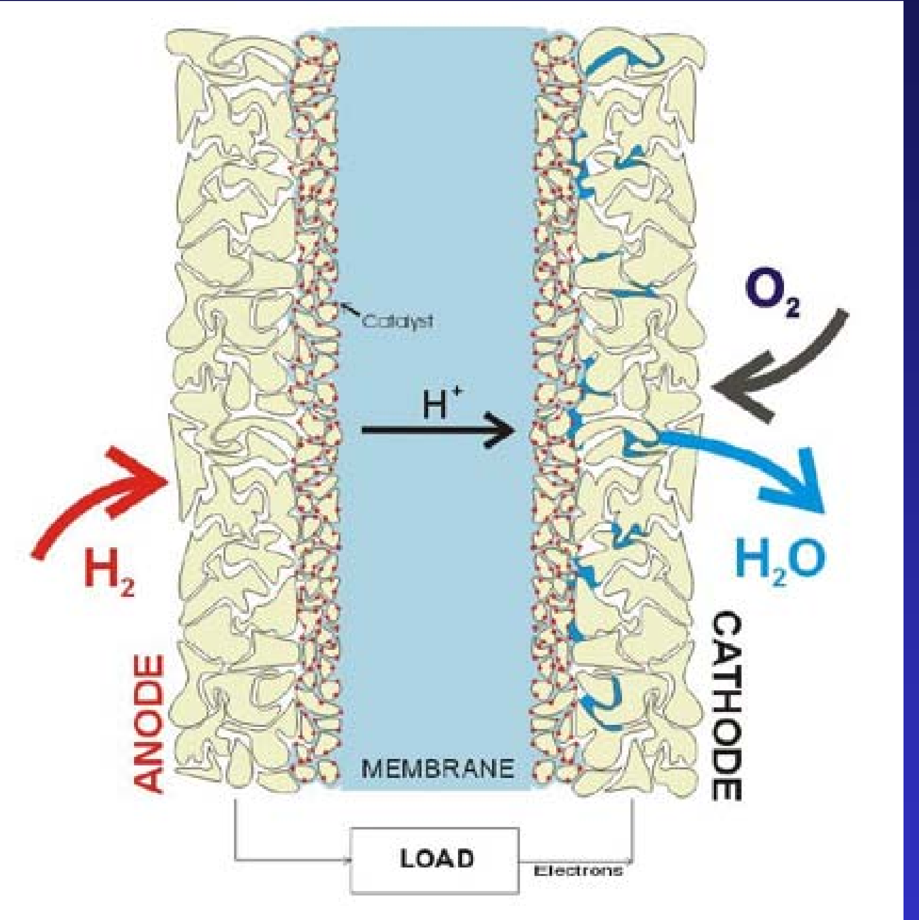
Mouse brain vasculature, PU-cast, resolution 2.5 µm





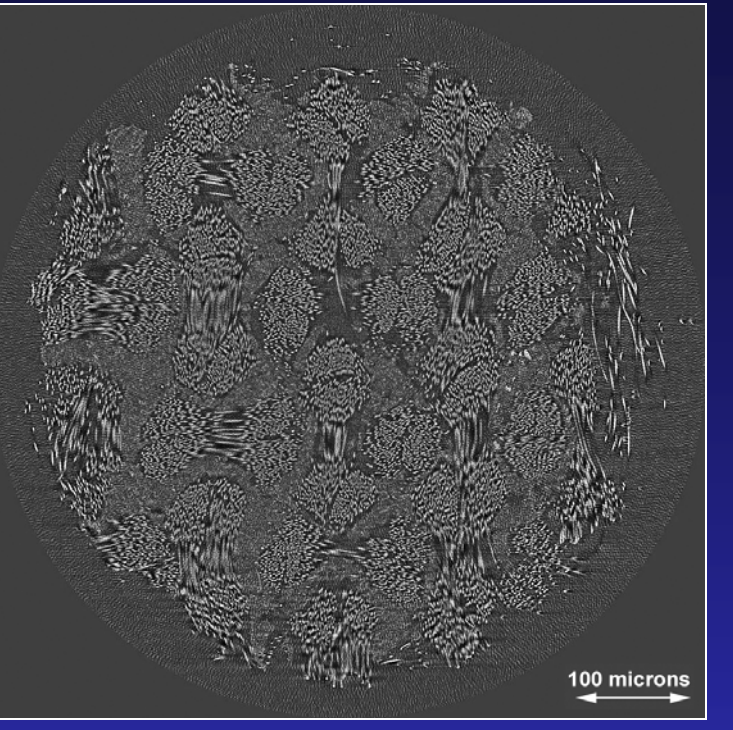


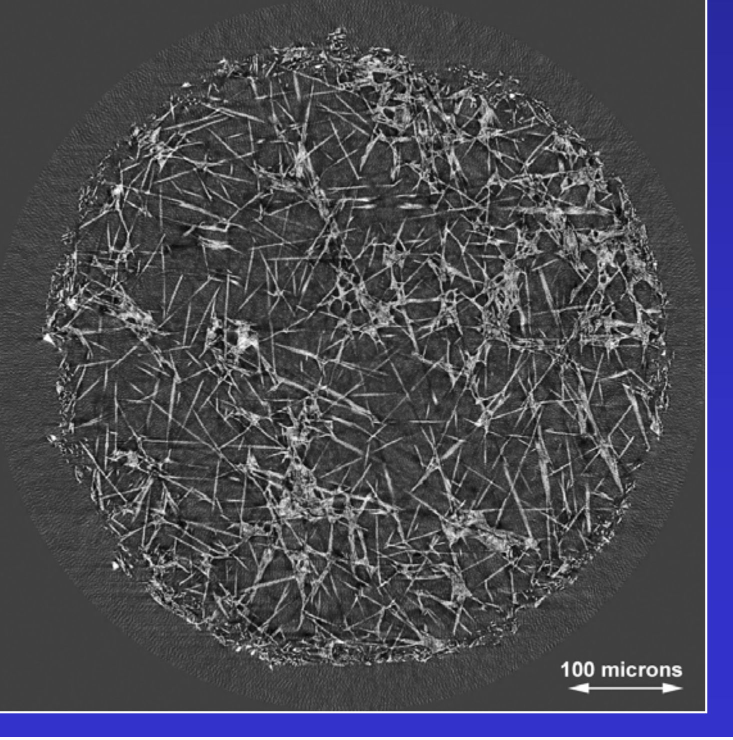
Energy research at PSI



Anode reaction $H_2 \rightarrow 2H^+ + 2e^-$

Cathode reaction $\frac{1}{2} O_2 + 2H^+ + 2e^- \rightarrow H_2O$





Polymer electrolyte fuel cells electrode

Courtesy of B. Andreaus and G. Scherer, PSI, Switzerland

SLS

Standard detector efficiency: 4-stages cascaded system

η_i = **gain (ratio of outgoing quanta to incoming quanta)**

$v_i = 1/\eta_i$ **for** $\eta_i >> 1$ **, Poisson distribution**

$v_i = (1-\eta_i)/\eta_i$ **for** $\eta_i < 1$ **, Binomial distribution**

$$DQE = \left(1 + v_1 + \frac{1}{\eta_1} \cdot v_2 + \frac{1}{\eta_1 \eta_2} \cdot v_3 + \ \ldots \right)^{-1} =$$

$$= \left(1 + \frac{1-\eta_{abs}}{\eta_{abs}} + \frac{1}{\eta_{abs}} \frac{1}{\eta_{flu}} + \frac{1}{\eta_{abs}\eta_{flu}} \frac{1-\eta_{col}}{\eta_{col}} + \frac{1}{\eta_{abs}\eta_{flu}\eta_{col}} \frac{1-\eta_{v/e}}{\eta_{v/e}} \right)^{-1}$$

Process	η_i			ν_i			DQE [%]		
Energy [keV]	10	20	30	10	20	30	10	20	30
Scint. abs. η_{abs}	0.08	0.07	0.02	11	13	40	8.44	7.00	2.45
Fluor. eff. η_{flu}	177	354	532	6×10^{-3}	3×10^{-3}	2×10^{-3}	8.40	6.98	2.44
Light coll. η_{col}		0.032			30.00		7.19	6.43	2.31
CCD QE $\eta_{\nu/e}$		0.22			3.54		4.71	5.01	1.94

Table 4.2: *Estimated DQE of a cascade system at 10, 20 and 30 keV for 5 μm YAG:Ce, NA = 0.7 and CCD quantum efficiency 22%.*

Limits of the standard detector

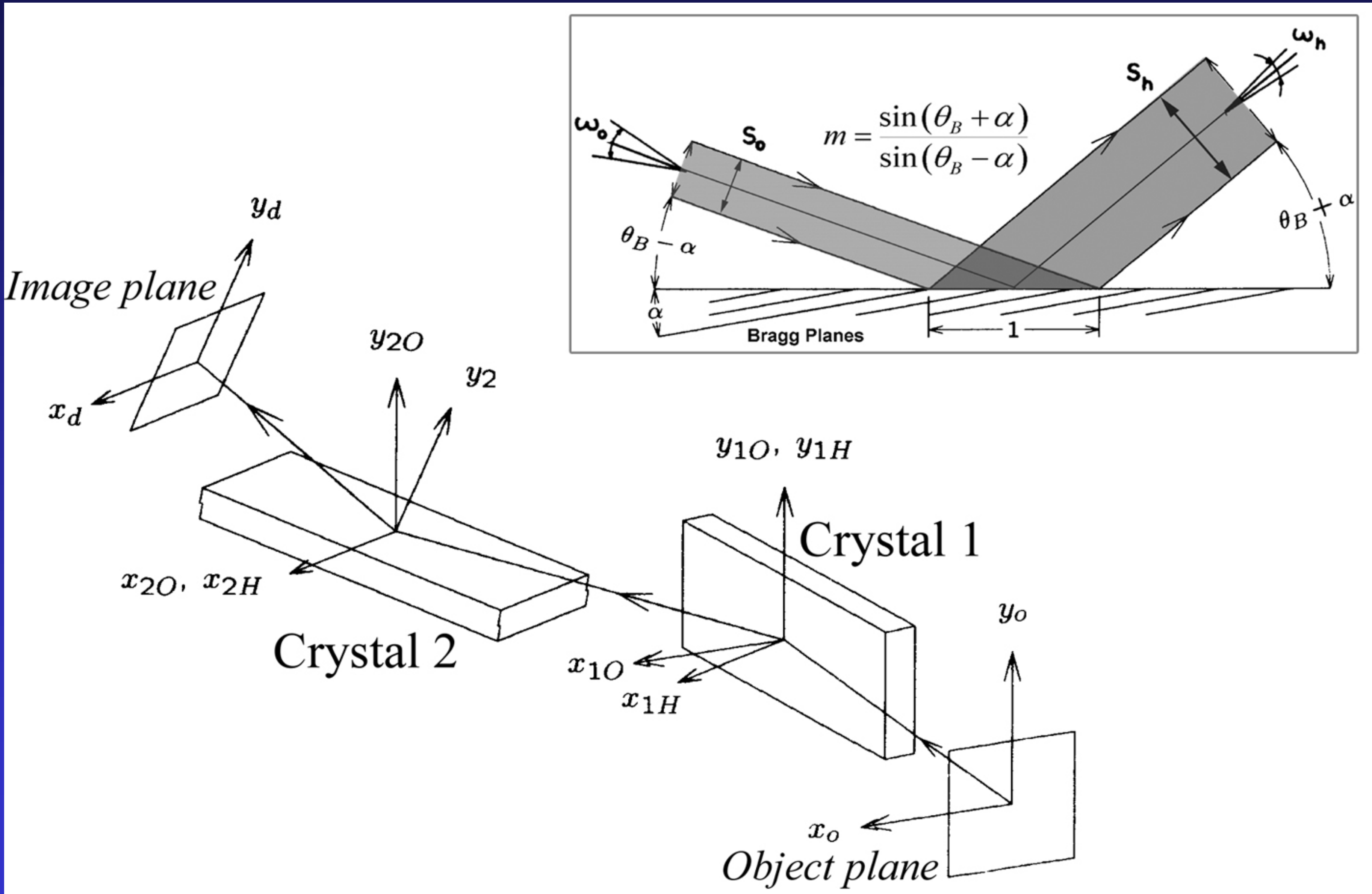
- Spatial resolution is directly coupled to the thickness of the scintillator.

- Resolution around 1 micron or below can be reached only to the detriment of the efficiency, i.e. using very thin scintillators.

- How to trespass the one micrometer barrier *efficiently*?

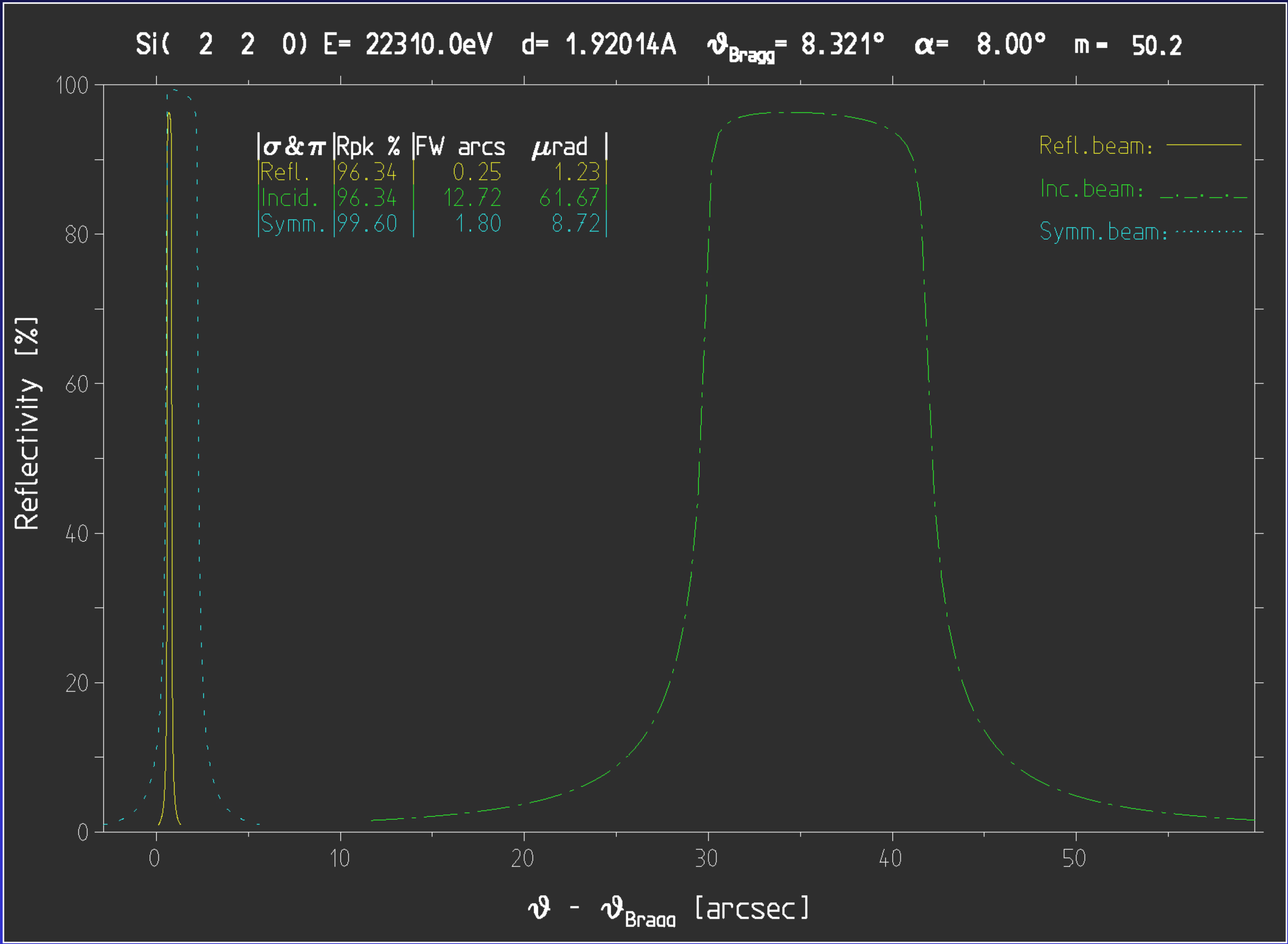
→ The solution is the „Bragg Magnifier“ !

Working principle of the Bragg Magnifier



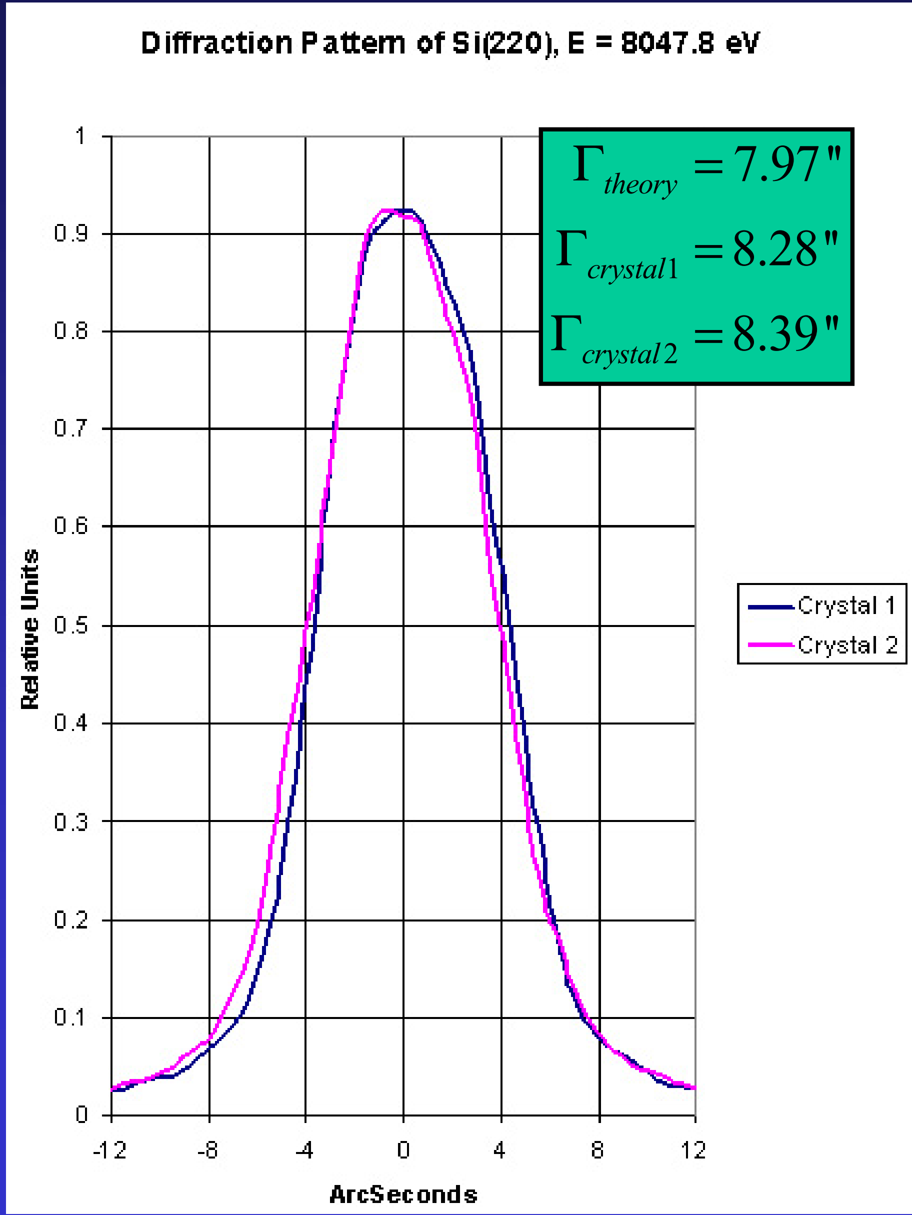
M. Stampanoni *et al.*, Proc. of SPIE, Vol. 5195, to be published

Diffraction properties of Si220

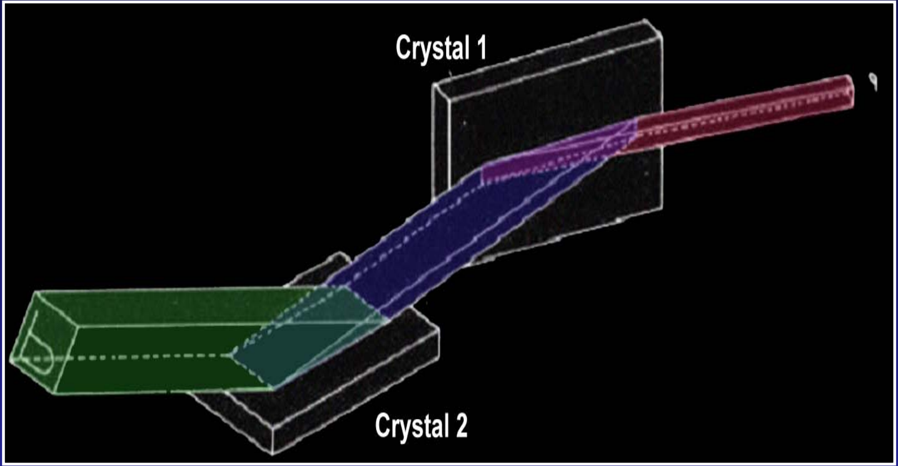


X-Ray Optics: cutting, polishing and characterizing...

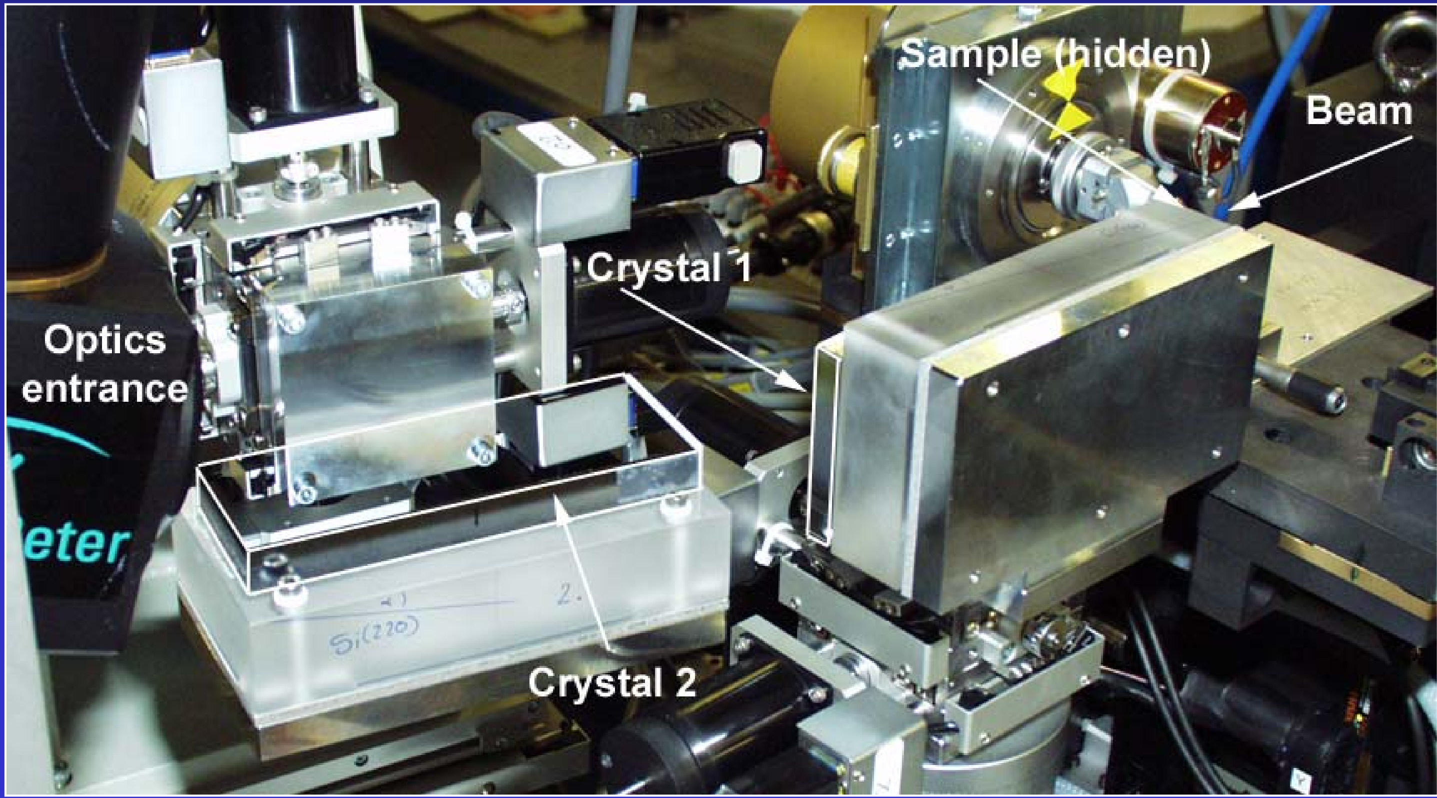
- Raw-Si material from **Wacker**
- Asymmetrical cut at **Holm** (Thann)
- At the **Institut für Kristallzüchtung** in Berlin:
  - topographic analysis(alignement)
  - fine asymmetrical cut (±0.001°)
  - polishing (Cyton-method)
- Fine polishing and optical mounting of Si-equivalent glass support at **Zeiss** (Oberkochen)
- Exp. characterization at PSI (Villigen)



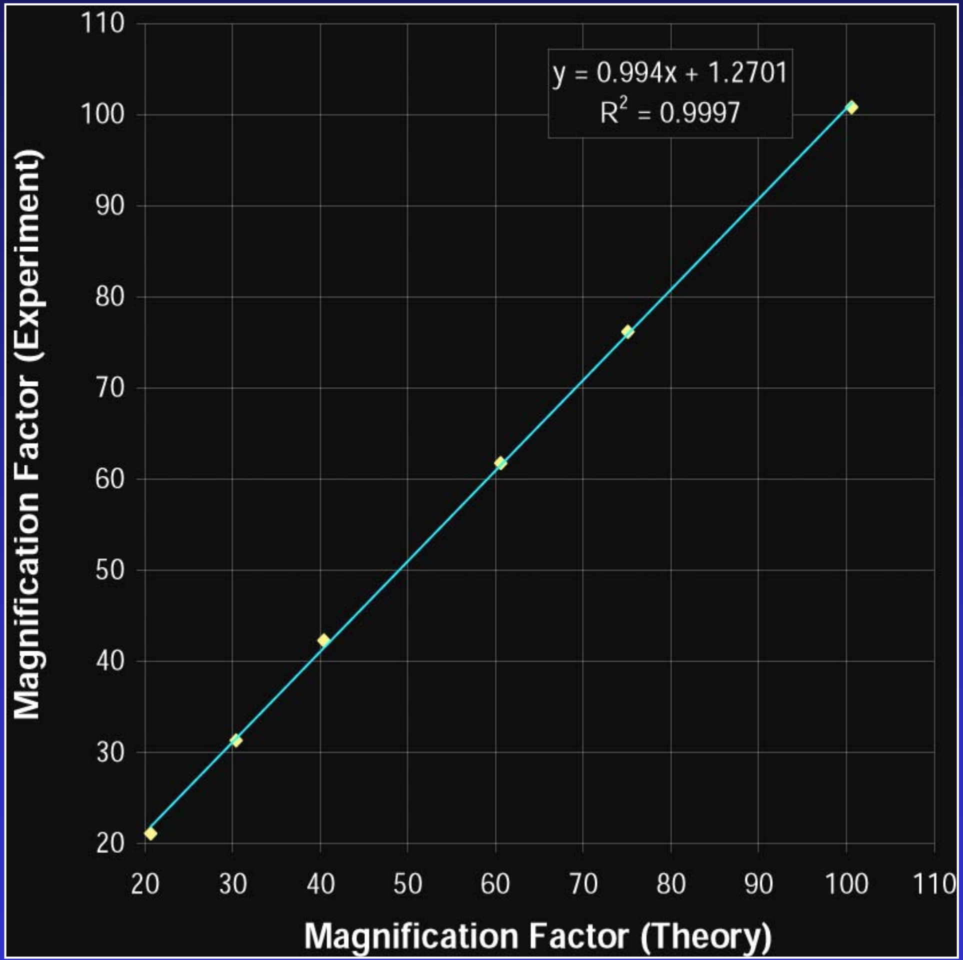
Bragg Magnifier at the MS beamline

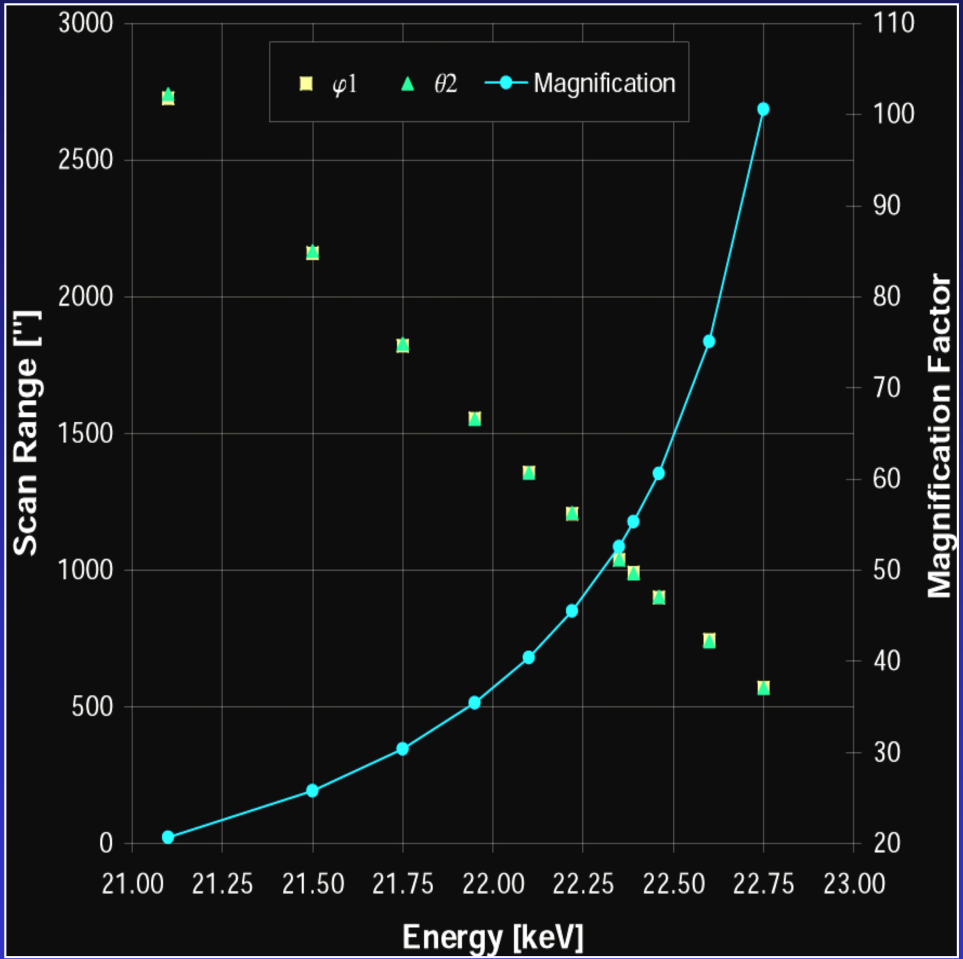


- Asymmetrical diffraction from Crystal 1 produces one-dimensional magnification
- A successive diffraction from Crystal 2, with the same magnification factor but perpendicular diffraction planes, produces uniform two-dimensional magnification



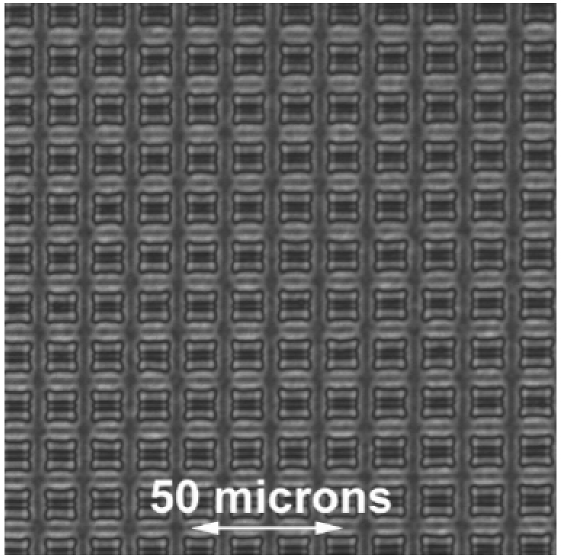
Reproducibility and reliability



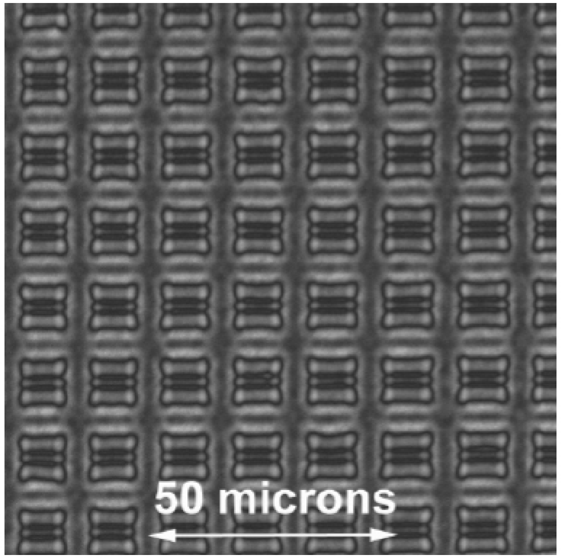


M. Stampanoni et al., Journal of Applied Physics, Vol. 92, Issue 12, 2002

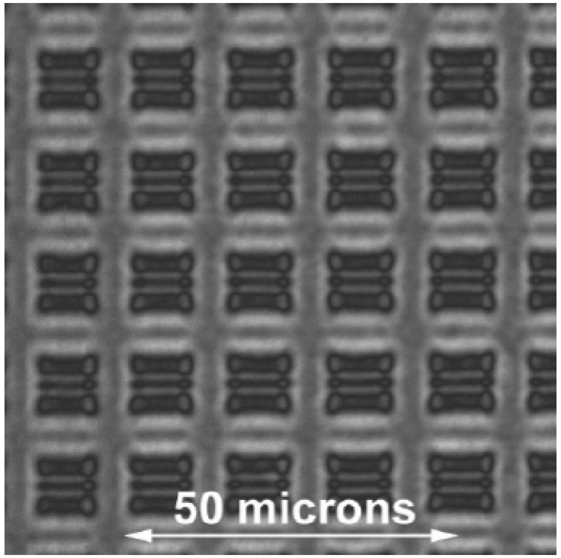
Bragg Magnifier in action



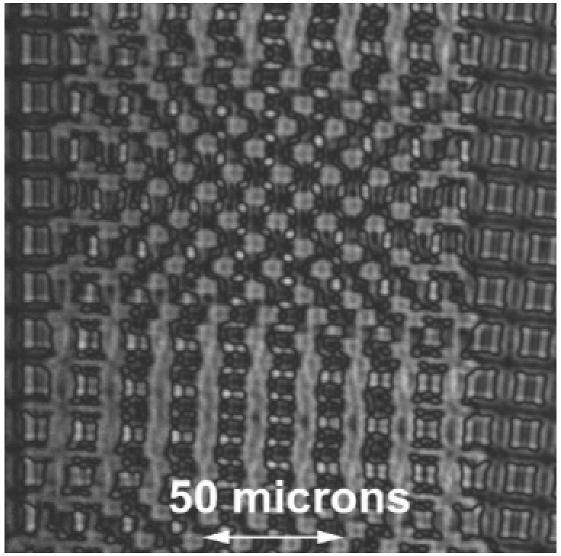
(a) Au mesh, magnification 40x40



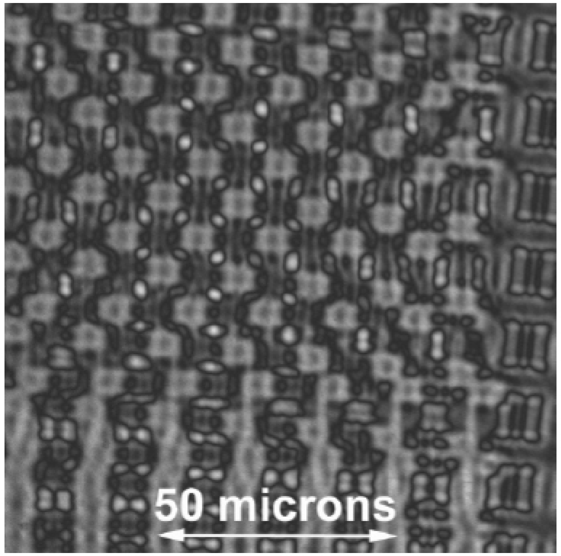
(b) Au mesh, magnification 60x60



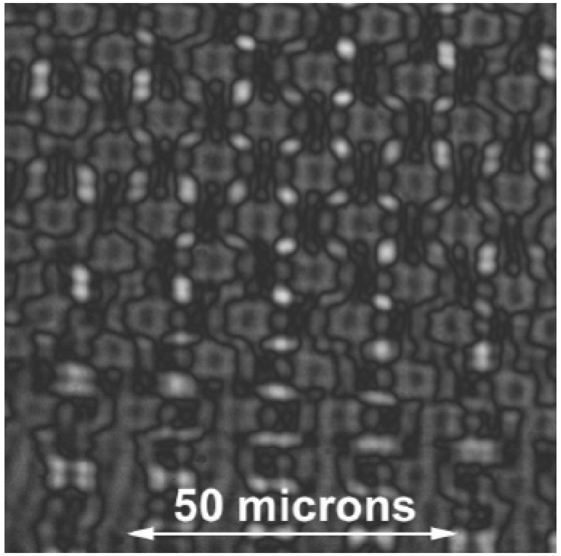
(c) Au mesh, magnification 80x80



(d) Titled mesh, magnification 40x40



(e) Titled mesh, magnification 60x60

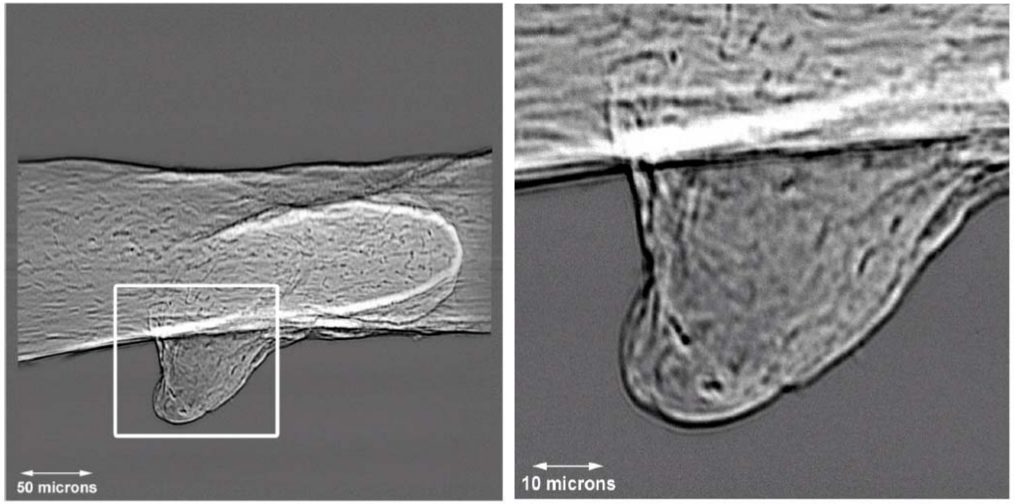


(f) Titled mesh, magnification 80x80

M. Stampanoni *et al.*, Proc. of SPIE, Vol. 5195, to be published

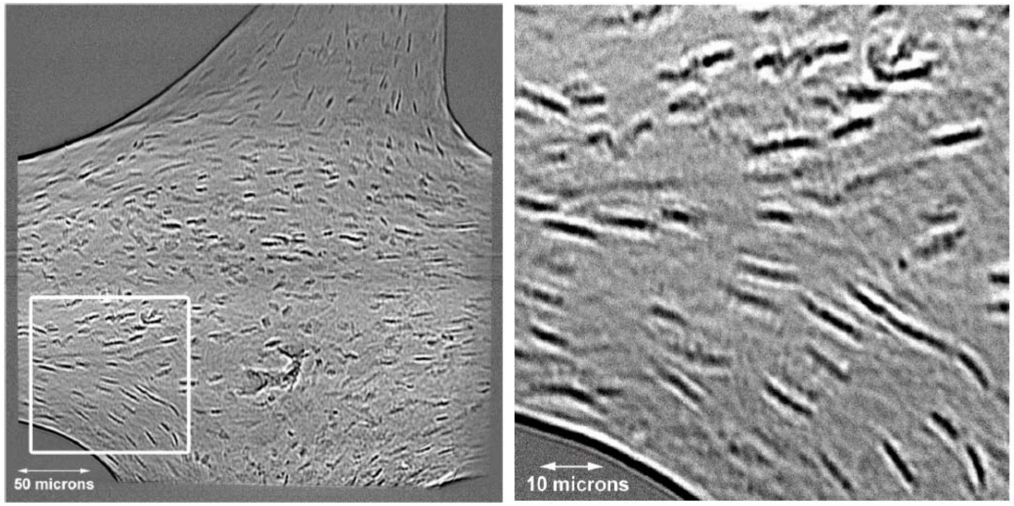
Example from medicine: Human Bone Trabecula

High resolution radiography



(b) Human trabecula imaged at 50x50.

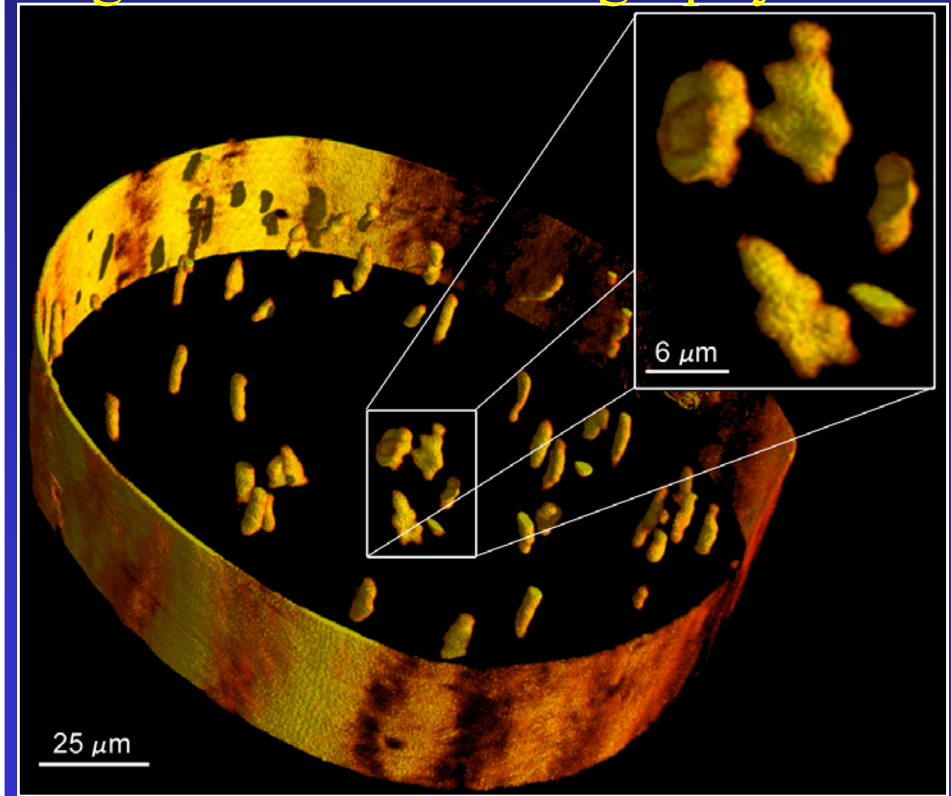
(c) Detail showing Fresnel patterns.



(d) Same trabecula, rotated by 90°.

(e) Detail showing osteocytes.

High resolution tomography



M. Stampanoni *et al.*, Applied Physics Letters, Vol. 82 (17), 2003

Advantages of the Bragg Magnifier

- Magnification in the X-ray range (transmission > 90%)
- Efficient (>90% at 22 keV) X-ray to visible light conversion without deterioring spatial resolution (thick scintillator)
- Magnification factors from 20x20 up to 100x100 can be easily achieved, for ENERGIES ABOVE 20 keV!!
- Image quality is improved because any scattering from the sample is eliminated due to the small angular acceptance of the crystals.

Data acquisition….

SLS File Server

Experiment

RAW DATA
(2048x2048)
(8 Mb/proj)

PRE-PROCESSING on the fly
Flat and dark field corrections
Sinograms generation
(16 Mb/sino)

End of measure

LINUX CLUSTER (20 GHz CPU)
slice of 2048x2048, 1000 proj
5.5 seconds per slice

RECONSTRUCTED DATA
650 slices per hour !!
(32 Mb/slice)

And from here science continues!!!
3D visualization, quantitative analysis...

Conclusions and Outlook

- Standard detector:
  - spatial resolution in the 1-5 μm range
  - soft as well as at hard X-ray energies
  - absorption and edge-enhanced imaging

- *Bragg Magnifier:*
  - novel development
  - spatial resolution well below one micrometer
  - high efficiency

- Quantitative 3D phase imaging
- Enhance the energy range of the Bragg Magnifier
- Automatic sample changer / sample alignment
- August 1-2, 2003: First user with Bragg Magnifier. Feedback is good.
- First test of the PILATUS pixel detector: very promising results!!

Last but not least: Development of a new, imaging dedicated beamline!

Many thanks to the following people:

SLS:
Rafael Abela, Bruce Patterson, Detlef Vermeulen, Steven Hunt, Renata Krempaska, Alain Bertrand, Marcel Grunder, Michael Lange, Timo Korhonen, Ahed Al-Adwan

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Peter Rüegsegger, Ralph Müller, Philipp Thurner, Philipp Schneider, Thomas Kohler, Paul Lüthi

TUM: Gunther Borchert

EMPA: Peter Wyss, Hugo Huber, Daniel Rechenmacher, Rolf Brönimann, Urs Sennhauser

THANK YOU!